DEVELOPMENT OF MULLITE FIBERS FOR HIGH-TEMPERATURE STRUCTURAL APPLICATIONS

FINAL PROGRESS REPORT

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13. ABSTRACT ***(Maximum 200 words)***

Inviscid Melt Spinning (IMS) has been shown to be appropriate for the production of mullite fibers. The formation of novel mullite fiber and the optimum processing conditions required to produce the fiber are described in two archival journal publications by Zhijun Xiao and the principal investigator. The development of a novel crucible design that was used to eliminate volatilization of SiO2 from the mullite melt and a corresponding change in melt composition is also described in a publication and a provisional patent.
Dr. Haoyue Zhang developed models and techniques for the characterization of crystallization kinetic parameters in these types of ceramic materials. The large-scaled production of mullite fiber and evaluation of the high-temperature properties of the fiber are the goals of future work. Work remains to be done in order to determine the suitability of IMS mullite fibers for high temperature structural composites.

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2. TABLE OF CONTENTS

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3. LIST OF APPENDIXES, ILLUSTRATIONS AND TABLES

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4. BODY

A. Statement of the Problem

Ceramic-matrix composites (CMCs) are being studied for high-temperature applications such as engine turbine blades, as they offer excellent performance/weight ratios. The reinforcing fiber in these CMCs is currently the focus of much research. Carbon, silicon carbide and sapphire are examples of fiber chemistries under consideration and testing for high-temperature CMCs. Each of these filaments has its drawbacks. An alternative fiber is proposed here; one composed of mullite. Mullite is a solid-solid solution of approximate composition 72 wt% Al_2O_3 and 28 wt% SiO_2. With a melting point of approximately 1880 - 1900°C, mullite offers excellent temperature resistance. It also possesses excellent mechanical and creep properties.

Mullite fibers are difficult to fabricate, however. Chemical vapor deposition and sol-gel techniques are diffusion-limited and slow. In addition, these techniques do not afford a great deal of control over the final fiber geometry. Inviscid Melt Spinning (IMS) is a fiber-forming technique that allows fibers to be formed directly from the melt. In the IMS process, the material to be fiberized is melted and extruded through an orifice into a reactive atmosphere. In low viscosity molten systems, such as high Al_2O_3-content aluminosilicates, the viscosity is not high enough to forestall Rayleigh breakup prior to stream solidification, and droplets, or shot, are formed instead of fiber. Inviscid systems are stabilized in the reactive atmosphere, however, and fiber can be produced through formation of a solid, stabilizing layer on the surface of the molten stream. In the case of mullite, the stabilizing material is expected to be carbon, the result of pyrolysis of propane in the vicinity of the molten jet. This project involves the development of IMS mullite fibers.

B. Summary of Results

The seminal goal of this project, namely, to demonstrate the viability of IMS as a method for producing mullite fibers, was met. As described in detail in the 1998 progress report, experimental quantities of mullite fibers were produced using an Inviscid Melt Spinning apparatus. An example of the fiber produced in this experiment is shown in Figure 1.

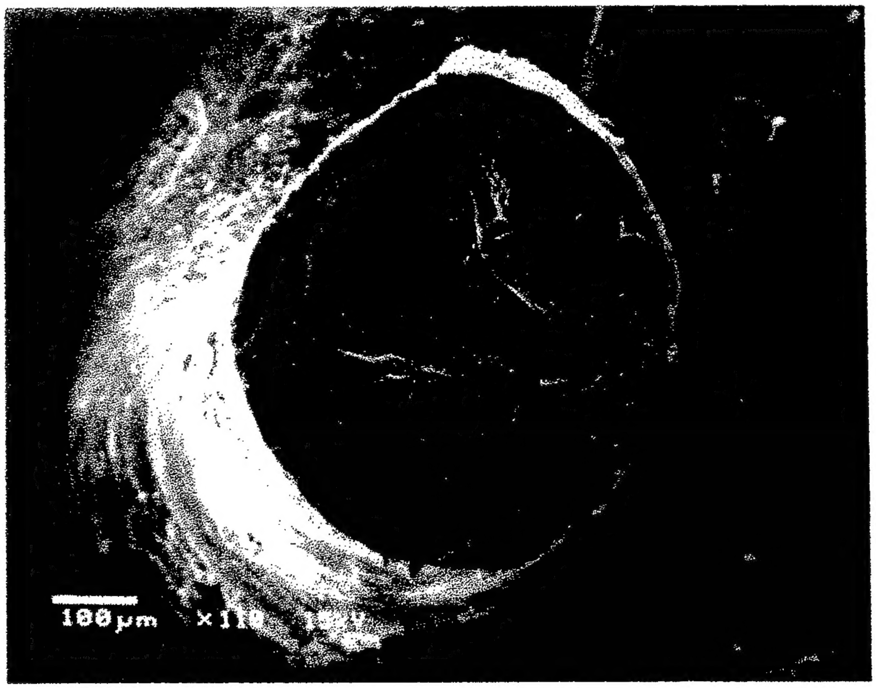

Figure 1 IMS Mullite Fiber.

The formation of this novel mullite fiber and the optimum processing conditions required to produce the fiber were described in two corresponding publications by Zhijun Xiao and the principal investigator [1,2]. This success did not come without a great deal of prior work, including the development of a novel crucible design that was used to eliminate volatilization of SiO_2 from the mullite melt and a corresponding change in melt composition. This design also led to a publication [3] and a provisional patent on the formation of mullite fibers via IMS [4]. The technical details of these accomplishments are described in the 1998 Progress Report and Dr. Xiao's thesis [5].

Since the last Interim Progress report, Dr. Haoyue Zhang, who was brought into the group to characterize the properties of ceramic fibers, developed some excellent models and techniques for the characterization of crystallization kinetic parameters in these types of materials [6]. It could well be that Dr. Zhang's new method for crystallization kinetics evaluation will be the single most important development of this project.

In summary, IMS has been shown to be appropriate for the production of mullite fibers. The large-scaled production of mullite fiber and evaluation of the high-temperature properties of the fiber are the goals of future work. Work remains to be done in order to determine the suitability of IMS mullite fibers for high temperature structural composites.

C. List of Publications

- Xiao, Z. and B.S. Mitchell, "Mullite Decomposition Kinetics and Melt Stabilization in the Temperature Range 1900-2000°C," *J. Am. Cer. Soc.*,83[4],761-767 (2000).
- Zhang, H.Y. and B.S. Mitchell, "A Method for Determining Crystallization Kinetic Parameters from One Non-isothermal Calorimetric Experiment," *J. Mat. Res.*, [15]4, 1000-1007 (2000).

- Xiao, Z. and B.S. Mitchell, "Optimization of Process Parameters in the Production of Mullite Fibers by Inviscid Melt Spinning," *Chem. Eng. Comm.*, [173], 123-133 (1999).
- Xiao, Z. and B.S. Mitchell, "The Production of Mullite Fibers by Inviscid Melt Spinning," *Mat. Lett.*, [37]6, 359-365 (1998).

D. List of Participating Scientific Personnel

Brian S. Mitchell, Ph.D. One month of summer salary in June, 1998, and June, 1999 for the principal investigator.

Haoyue Zhang, Ph.D. Postdoctoral scientist working on mullite fiber characterization. Terminated December, 1999.

Zhijun Xiao. Ph.D. received May, 1998. Primary developer of mullite IMS fibers.

Wenyan Li. M.S. received December, 1998. Assisted Dr. Zhang in crystallization studies.

Fernando Fondeur. Ph.D. received May, 1998. Assisted Z. Xiao with IMS experiments and fiber characterization.

Yinfeng Zong Ph.D. candidate, transferred to RPI, Fall, 1999.

Nick Maljkovic. M.S. anticipated June, 2000. Assisted Dr. Zhang.

Stacey Bennett. Undergraduate student working on DTA analysis. Graduated May, 2000.

Brian Smith. Undergraduate student working on equipment modification. Graduated May, 1999.

Mike Farley. Undergraduate student working on equipment modification. Graduated May, 1998.

Cory McGraw. Undergraduate student working on equipment modification. Graduated May, 2000.

5. REPORT OF INVENTIONS

- Inviscid Melt Spinning of Mullite Fibers, U.S. Provisional Patent, Filed May 28, 1999.

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1. Xiao, Z. and B.S. Mitchell, "The Production of Mullite Fibers by Inviscid Melt Spinning," *Mat. Lett.*, [37]6, 359-365 (1998).

2. Xiao, Z. and B.S. Mitchell, "Optimization of Process Parameters in the Production of Mullite Fibers by Inviscid Melt Spinning," *Chem. Eng. Comm.*, [173], 123-133 (1999).

3. Xiao, Z. and B.S. Mitchell, "Mullite Decomposition Kinetics and Melt Stabilization in the Temperature Range 1900-2000°C," *J. Am. Cer. Soc.*,83[4],761-767 (2000).

4. Inviscid Melt Spinning of Mullite Fibers, U.S. Provisional Patent, Filed May 28, 1999.

5. Xiao, Z., "Formation and Characterization of Mullite Fibers Produced by Inviscid Melt Spinning," Ph.D. thesis, Tulane University, 1998.

6. Zhang, H.Y. and B.S. Mitchell, "A Method for Determining Crystallization Kinetic Parameters from One Non-isothermal Calorimetric Experiment," *J. Mat. Res.*, [15]4, 1000-1007 (2000).

7. APPENDIXES

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